

| Data Acceptability Criteria for Organic Compounds in Tissue & Sediment (PCB's, PAH's, Pesticides);<br>and for Semi-Volatiles & Volatiles in Sediment Only  |   |  |  |  |
|--|---|--|--|--|
| Sample Type  | Objective   | Frequency of Analysis  | Recommended Control Limits   | Recommended Corrective Action  |
| <b>External Calibration</b><br>Calibration Standards (3-5 standards over the expected range of sample target analyte conc., with the lowest conc. Std at or near the MDL).   | Full calibration: Establish relationship between instrument response and target analyte conc.   | Follow manufacturer's or procedures in specific analytical protocols. A min., 3 point calib. Each set up, major disruption, and when routine calib check exceeds specific control limits.              | Linear regression, $r > 0.995$ .   | Determine cause and take appropriate corrective action. Recalibrate and reanalyze all suspect samples or flag all suspect data.  |
| <b>Calibration Verification</b><br>Calibration Check Standards (minimum of one mid-range standard: an instrument internal standard must be added to each calib. check std. when internal std. calib. is being used).                         | Verify calibration.   | After initial calibration or recalibration. Every 10 samples.  | %R = 85-115%.  | Determine cause and take appropriate corrective action. Recalibrate and reanalyze all suspect samples or flag all suspect data.  |
| <b>Method Detection Limit Determination</b><br>Spiked matrix samples (analyte-free tissue or sediment samples to which known amounts of target analytes have been added; one spike for each target analyte at 3-10 times the estimated MDL). | Establish or confirm MDL for analyte of interest.   | Seven replicate analyses prior to use of method. Re-evaluation of MDL annually   | Determined by program manager.   | Redetermine MDL.   |
| <b>Accuracy and Precision Assessment</b><br>Reference materials (SRMs or CRMs, prepared from actual contaminated fish or shellfish tissue or sediment if possible, covering the range of expected target analyte conc).                      | Assess method performance (initial method validation and routine accuracy assessment).  | Method validation: As many as required to assess accuracy and precision of method before routine analysis of samples. Routine accuracy assessment: one (preferably blind) per 20 samples or one batch. | Measured value 70-130% of the 95% confidence intervals, if certified. Otherwise, %Recovery = 50-150%.              | Failure of any of the accuracy and precision control limits require the following. Determine cause and take appropriate corrective action. Recalibrate and reanalyze all suspect samples or flag all suspect data. |
| Matrix spikes (composite tissue or sediment homogenates of field samples to which known amounts of target analytes have been added: 5 times the concentration of the analyte of interest or 10 times the MDL).                               | Assess matrix effects and accuracy (%Recovery) routinely.   | One per 20 samples or one per batch, whichever is more frequent.   | %Recovery = 50-150% or Control Limits based on 3x the standard deviation of laboratory's actual method recoveries. | See Reference Materials Corrective Action. Zero percent recovery requires rejection of all suspect data.   |
| Matrix spike replicates (replicate aliquots of matrix spike samples; 5 times the concentration of the analyte of interest or 10 times the MDL).  | Assess method precision routinely.  | One duplicate per 20 samples or one per batch, whichever is more frequent.   | RPD <25% for duplicates.   | See Reference Materials Corrective Action.   |
| Field Replicate (replicate aliquots of tissue and sediment field samples).   | Assess method precision routinely. Assess total variability (i.e., population variability, field or sampling variability, and analytical method variability.) | One field duplicate sample per 20 samples or one per batch, whichever is more frequent.  | RPD <25% for duplicates.   | Determine cause and take appropriate corrective action. Recalibrate and reanalyze all suspect samples or flag all suspect data.  |

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|---|---|---|---|--|
| <b>Sample Type</b>  | <b>Objective</b>  | <b>Frequency of Analysis</b>  | <b>Recommended Control Limits</b>                                   | <b>Recommended Corrective Action</b>   |
| <b>Contamination Assessment</b><br>Laboratory Blanks (method, processing, bottle, reagent).   | Assess contamination from equipment, reagents, etc.   | One method blank per 20 samples or one per batch, whichever is more frequent. At least one bottle blank per batch. One reagent blank prior to use of a new batch of reagent and whenever method blank exceeds control limits.   | Concentration of any analyte <MDL as determined by program manager. | Determine cause of problem (e.g., contaminated reagents, equipment), remove sources of contamination, and reanalyze all suspect samples or flag all suspect data.  |
| Field Blanks, Travel Blanks, Equipment Blanks.  | Assess contamination from equipment, from air, from surrounding environment, etc.           | One travel blank and one field blank is required per every 20 (or less) field samples collected for volatile organic analytes (VOC's, MTBE, BTEX) in sediment. No travel, field, or equipment blanks are required for other (non-volatile, semi-volatile) organic compound samples in sediment or tissue. | Blanks < MDL.   | Determine cause of problem (e.g., contaminated preservatives, equipment contamination, improper cleaning, exposure to airborne contaminants, etc.), remove sources of contamination, and reanalyze all suspect samples or flag all suspect data. |
| <b>Routine Monitoring of Method Performance for Organic Analysis</b><br>Surrogate Spikes (Prepared from chemicals of similar structure to target analytes or isotopically labelled target analyte). | Assess method performance and estimate recovery of target analytes analyzed by GC or GC/MS. | In every calibration standard, sample, and blank analyzed for organics by GC or isotope dilution GC-MS; added to samples prior to extraction.   | Determined by program manager.                                      | Determine cause of problem (e.g., incomplete extraction or digestion, contamination, inaccurate preparation of internal standard), take appropriate corrective action, and reanalyze all suspect samples or flag all suspect data.               |
| <b>External QA Assessment</b><br>Accuracy-based performance evaluation samples submitted to new laboratories by SWAMP QA Program.   | Initial demonstration of laboratory capability.   | Once prior to routine analysis of field samples.  | Determined by study manager.  | Determine cause of problem and reanalyze sample. Do not begin analysis of field samples until laboratory initial capability is clearly demonstrated.   |
| Mandatory interlaboratory exercises overseen by 3rd party external ("referee") SWAMP QA Program officials for all SWAMP participant laboratories.   | Ongoing demonstration of laboratory capability.   | One exercise per year.  | Determined by study manager.  | Determine cause of problem and reanalyze sample. Further corrective action to be determined by QA manager.   |
| Voluntary, but encouraged, participation in NOAA-NIST intercalibration studies and CA-ELAP annual performance evaluations, as appropriate.  | Ongoing demonstration of laboratory capability.   | One exercise per year.  | Determined by study manager.  | Determine cause of problem and reanalyze sample. Further corrective action to be determined by QA manager.   |
| <b>General Provisions</b><br>Acceptable Data Set: CCV Recoveries must be within control limits, & either SRM or Spiked Matrix recoveries must also be within control limits.                        |   |   |   |  |